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(54) **A polymeric blend useful in thin-bed mortar compositions.**

(57) This invention concerns a polymer blend having a water-soluble cellulose ether and a water-insoluble, but water-dispersible polymer which is useful in thin-bed mortar compositions. The water insoluble, but water-dispersible polymer can include an unneutralized polyacrylic acid or methylcellulose with a viscosity from 50 cps to 150,000 cps. The polymeric blend extends the working time of the composition in which it is employed and because the blend has sag binding characteristics, it also functions as a sag resistance aid.

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Description

A POLYMERIC BLEND USEFUL IN THIN-BED MORTAR COMPOSITIONS

Many adhesives and cementitious compositions such as construction mortars, concretes and plasters are typically formulated with a water-retention aid to improve the characteristics thereof. By employing a water-retention aid, water loss is prevented and the rheological properties are improved. Typically, water-retention aids are used in ceramic tile adhesive compositions to prevent water absorption by the ceramic tile or substrate to which the tile is applied.

To be functional in the construction industry, a ceramic tile composition must be easily spreadable and in conjunction the composition must support the tile during the period in which the composition sets. Typically, a thin-bed mortar composition used to set ceramic tile with a preference for thin-bed mortars contains: sand, cement, and cellulose ethers. It is known that cellulose ethers extend the working time of the thin-bed composition, acting as a water-retention aid.

Unfortunately, cellulose ethers do not contribute to sag resistance, or resistance to movement of the tile that is applied to the thin-bed mortar composition on a vertical surface. However, as disclosed in U.S. Patent 4,487,864 when cellulose ethers are crosslinked to form a modified water-soluble carbohydrate, the carbohydrate is useful as a water-retention aid and as an adhesive.

Another method of controlling sagging is disclosed in U.S. Patent 4,021,257 in which the use of long chain organic polymers, which function as flocculants, aids in sag resistance. In practice, typically, the most operable compositions are mixtures of clays with cellulose ethers and polyacrylamide flocculants, but these are costly and have handling disadvantages.

Thus, it would be highly desirable to have a composition which has all three properties - good rheology, good bond characteristics, has water-retention properties - and can be used efficiently without cost and handling disadvantages.

This invention concerns a polymer blend useful in thin-bed mortar compositions which comprises a water-soluble cellulose ether with a viscosity from 2,000 to 100,000 cps and a water-insoluble, water-dispersible polymer with a viscosity from 50 to 150,000 cps. The water-insoluble, water-dispersible polymer can include an unneutralized polyacrylic acid, that is lightly crosslinked, or a cellulose ether such as methylcellulose.

Generally, this polymeric blend can be used in ceramic tile mortars, cementitious compositions, wall-finishing compounds, cement plasters, and in tape joint compounds. The polymeric blend extends the working time of the composition, since the blend has water retention properties. Thus, the composition, e.g., mortar composition, is easy to use and thin-bed adhesives are possible. By using a water-insoluble, water-dispersible polymer in the polymeric blend, the blend has strong binding characteristics and it also functions as a sag resistance aid.

By "water-insoluble, water-dispersible" is meant that the polymer can be water-swellable, due to the level of or type of substitution and is dependent upon the pH of the medium in which the polymer is employed. Typically, the polymer will function as water-swellable in a pH of 12 or greater, but the pH is not a limiting factor. The polymer will absorb at least 50, preferably 2,000 grams (g) of water per gram of polymer. In addition, the water-insoluble, water-dispersible polymer can include certain cellulose ethers that are water-swellable, or a crosslinked polymer such as a polyacrylic acid, which is also water-swellable due to the crosslinking.

To function as a water-insoluble, water-dispersible polymer, the crosslinked polymer must be insoluble in water and be unneutralized, even though in the process of utilizing the blend in mortar compositions and the like, the acid does become neutralized. For example, acrylic acid in the acid form rather than in the salt form preferably is employed as the water-dispersible polymer. Thus, water-dispersible polymers can include crosslinked polymers such as polyacrylic acids or water-swellable polymers such as certain cellulose ethers.

The water-insoluble, water-dispersible cellulose ether is inherently water-swellable and can include a methylcellulose. The water-swellability is the greatest at a pH of 12 or greater, although the water-insoluble, water-dispersible cellulose ether can be water-swellable at a pH below 12 depending upon the methoxyl content. Additionally, the water-swellable cellulose ether (at a pH below or above 12) has a methoxyl content from 10 to 25 weight percent, preferably from 18 to 23 weight percent; and has a hydroxypropyl content from 0 to 6 weight percent, preferably from 0 to 4 weight percent. Most preferably, the hydroxypropyl group is not present and only the methoxyl group is present at a concentration of 18 to 23 weight percent.

At a pH below 12, the cellulose ether has a viscosity from 50 to 50,000 centipoises (cps). Preferably, the cellulose ether has a viscosity from 100 to 40,000 cps, and most preferably from 500 to 30,000 cps, as a 2 weight percent solution in water at 20°C. In a pH of 12 or greater, the cellulose ether swells much more than at a pH below 12, and the viscosity of the cellulose ether is from 500 to 150,000 cps. Preferably, the cellulose ether has a viscosity from 1,000 to 100,000 cps, and most preferably from 2,000 to 50,000 cps, as a 2 weight percent solution in water at 20°C.

The polyacrylic acid usable in the present invention has a pH less than 7 and is comprised of a copolymer of a vinyl acrylic acid monomer that contains from 0.05 to 2 percent crosslinking agent. The vinyl acrylic acid monomer can include, for example, acrylic acids, methylacrylic acids, or their copolymers, and the crosslinking agent can include, for example, trimethyolpropane triacrylate. Standard polymerization techniques known in the art may be used to crosslink the monomer to form the polymer. Typically, 0.10 to 0.50 percent of the

crosslinking agent is combined with the crosslinkable monomer. The monomer must be crosslinked, forming a polyacrylic acid, before employing the polyacrylic acid in mortars, cement plasters and the like.

In addition, the water-swellable polymer must be non-flocculating, meaning a material that does not induce agglomeration of particles present in cementitious compositions, mortars, adhesives and the like. Flocculation is determined by the method described in U.S. Patent 4,021,257. According to the procedures described in U.S. Patent 4,021,257, column 5, a polymer is non-flocculating if its flocculation index is greater than 0.50, preferably 0.60, more preferably 0.70. The water-swellable cellulose ethers used herein have a flocculation index of 0.78 and the polyacrylic acid has a flocculation index of 0.65; therefore, both are non-flocculants.

The water-soluble cellulose ether can be, for example, hydroxypropyl methylcellulose, methylcellulose, ethylhydroxyethylcellulose, hydroxyethylmethylcellulose or hydroxyethylcellulose, and have a viscosity from 2,000 to 100,000 cps, preferably from 4,000 to 50,000 cps. A suitable water-soluble methoxyl content is from 16 to 32 weight percent, preferably from 19 to 24 weight percent and the hydroxypropyl content is from 4 to 32 weight percent, preferably from 7 to 12 weight percent.

The polymeric blend may be employed in various ratios to impart the desired rheological properties. Generally, from 3:1 to 1:3 by weight, respectively, water-soluble cellulose ether to water-insoluble, water-dispersible cellulose ether is operable, and the preferable ratio is from 2:1 to 1:2 by weight of the water-soluble cellulose ether to water-insoluble, water-dispersible cellulose ether. Most preferably, the ratio of water-soluble polymer to water-insoluble, water-dispersible polymer is about 1:1. Generally, the polymeric blend is preblended as a dry powdery mix in the said ratios. The polymeric blend may be added to other ingredients commonly used in forming cementitious or adhesive compositions and the like. However, all the ingredients can be added simultaneously forming the desired composition.

Typically, the polymeric blend of the invention can be employed in ceramic tile mortars, in mortar compositions, wall-finishing compounds, crack fillers, cement plasters, and in tape joint compounds. As used herein, cementitious compositions refer to hydraulic cement and cement is meant to include materials which in admixture with water form a hydrate. Common cements can include alumina cement, Portland cement, and gypsum.

These thin-bed mortars and cementitious compositions must have strong bonding characteristics known as sag resistance, and additionally, must have water-retention properties to ensure suitable rheology of the composition. Accordingly, in a further aspect of the invention, there is provided a mortar composition comprising a water-soluble cellulose ether having a viscosity of from 2,000 to 100,000 cps, and a water-insoluble water dispersible polymer with a viscosity of from 50 to 150,000 cps wherein the total amount of the said cellulose ether and polymer is from 0.2 to 2 percent by weight of the dry components of the mortar. Typically, these water-soluble/-water-insoluble, water-dispersible thickener components constitute from 0.2 to 2 percent, preferably from 0.2 to 1.0 weight percent of the mortar cementitious composition based on dry components. Preferably, the thickener components made up from 0.4 to 0.7 weight percent thereof.

In particular, a typical ceramic tile adhesive composition comprises from 25 to 97 weight percent Portland cement, from 0 to 75 weight percent filler and from 0.2 weight percent to 2 weight percent water-soluble/water-swellable polymeric blend. The inert filler is optional, but if desired can include sand limestone, talc, mica, silica, or most any other inert particulate inorganic material.

Also of interest are cementitious compositions, cement plasters or stucco compositions. Cementitious or stucco compositions can comprise from 50 to 80 weight percent Portland cement, from 20 to 50 weight percent filler such as sand, and from 0.2 to 0.5 weight percent water-soluble/water-insoluble polymeric blend, based on cement. Optionally, surfactants may be added to improve rheology characteristics.

When preparing a gypsum-plaster composition, the composition is similar to the cementitious compositions except 3 to 4 weight percent of the composition is limestone and about 0.2 weight percent is the water-soluble/water-insoluble polymeric blend. Whereas, when preparing a ceramic tile adhesive composition, the composition is similar to the cementitious composition except about 0.5 weight percent of the water-soluble, water-insoluble polymeric blend is used based on cement.

Additionally, the amount of water used with the composition will depend somewhat in the particular use and desired consistency for which the composition is to be employed.

The following examples are provided to illustrate the invention.

EXAMPLES

These mixtures are non-flocculating, meaning a material which does not substantially induce agglomeration of particles. A method for determining flocculation activity (flocculation index) is the test described in U.S. Patent 4,021,257. Water-insoluble, water-dispersible organic polymers having a flocculation index, as measured by the test in the patent, of greater than 0.50, may be considered non-flocculants for the purpose of this invention. Polyacrylic acid gives a flocculation index of 0.65 and the methylcellulose used in the said invention gives a flocculation index of 0.78. Thus, the water-insoluble, water-dispersible polymers used herein are non-flocculating.

The examples in Tables I and II are prepared in the following manner:

Dry Ingredients:

A dry-set cement mortar composition is prepared by blending the dry ingredients as follows:

- 50.0 percent graded Ottowa Sand

- 49.5 percent Portland Cement
- 0.5 percent total amount of water-soluble/water-swellable polymeric blend*
(*the ratio of water-soluble and water-swellable polymer is specified in Table I)

Water Ratio:
To ensure proper consistency for spreading, 22.0 g of water are added to 100 g of the dry cement blend.

Mixing Mortar:
The mixing operation of mortar with water should be performed slowly until uniform mixing is achieved, and then vigorously, for proper air entrainment and consistency. The mixing operation should be performed 5 to 6 minutes after the initial water addition. Shake for 30 minutes and remix before using.

THICKENER SYSTEM IN TABLE I - varying ratios of the hydroxypropyl methylcellulose and polyacrylic acid in the blend to show the effects thereof.

1. The thickening system comprises hydroxypropyl methylcellulose and polyacrylic acid (Carbopol® 934, available from the B. F. Goodrich Company). The water-soluble cellulose ether has a viscosity of 5,337 cps as a 2 percent solution and a methoxyl content of 22.1 weight percent with a hydroxypropyl content of 8.1 weight percent to about 12 weight percent. The polyacrylic acid is Carbopol® 934 and has a viscosity of about 416,000 cps at 0.5 percent solids using a Brookfield RVT Viscometer with a number 5 spindle at 2.5 rpm.

2. The same as in 1, except the ratio of water-soluble cellulose ether to water-insoluble, water-dispersible polymer is 1.7:1.

3. The same as in 1, except the ratio of water-soluble cellulose ether to water-insoluble, water-dispersible polymer is 1:1.

4. The same as in 1, except the ratio of water-soluble cellulose ether to water-insoluble, water-dispersible polymer is 1:3.

THICKENER SYSTEM IN TABLE II - varying the methoxyl content of the water-dispersible methylcellulose, but also altering the water-soluble polymer.

5. The thickening system comprises: hydroxypropyl methylcellulose and methylcellulose. The water-soluble cellulose ether, hydroxypropyl methylcellulose has a viscosity of 5337 cps as a 2 percent solution and has a methoxyl content of 22.1 weight percent with a hydroxypropyl content of 8.1 weight percent. The water-insoluble, water-dispersible polymer, methylcellulose, has a viscosity of 2,500 cps as a 2 percent solution and has a methoxyl content of 16.7 weight percent with no hydroxypropyl present.

6. The water-soluble cellulose ether has a methoxyl content of 31.4 weight percent with no hydroxypropyl present, and has a viscosity of 3,949 cps as a 2 percent solution. The water-insoluble, water-dispersible polymer, methylcellulose, has a methoxyl content of 22.5 weight percent with no hydroxypropyl present and has a viscosity of 7,500 cps as a 2 weight percent solution.

7. The same as in 6, except that the water-dispersible cellulose ether contains 16.7 weight percent methoxyl with no hydroxypropyl present, and has a viscosity of 2,500 cps as a 2 percent solution.

8. The same as in 6, except that the water-dispersible methylcellulose has a methoxyl content of 16.8 weight percent with no hydroxypropyl present and the methylcellulose has a viscosity of 300 cps as a 2 weight percent solution.

The Examples Have the Following Qualities:
The compositions in Tables I and II have good rheology and when applied to tiles, there is excellent support; thus the composition has good bonding characteristics. In addition, the compositions retain water sufficiently to allow the mixture to have extended working time.

CONTROLS

A. The control comprises an unneutralized polyacrylic acid Carbapol® 934 with a viscosity of 416,000, at 0.5 percent solids using a Brookfield RVT Viscometer with a number 5 spindle at 25 rpm as a 0.5 weight percent solution. The control has no water retention properties and has extremely poor workability properties.

B. The control comprises hydroxypropyl methylcellulose with a viscosity of 5,337 cps as a 2 percent solution. The hydroxypropyl methylcellulose has a methoxyl content of 22.1 weight percent and a hydroxypropyl content of 8.1 weight percent.

TABLE I

Sample No.	Thickener System		Ratio of Water-Soluble Cellulose Ether to Water-Insoluble, Water-Dispersible Polymer	Sag Resistance Value** (mm)
	Water-Soluble Cellulose Ether	Water-Insoluble, Water-Dispersible Polymer		
1	Hydroxypropyl methylcellulose	Polyacrylic acid	3:1	10.0
2	"	"	1.7:1	3.0
3	"	"	1:1	2.0
4	"	"	1:3	0
A	0	*polyacrylic acid	0:1	N/A

*Control - See page 12
**See page 15

TABLE II

Sample No.	Thickener System		Ratio of Water-Soluble Cellulose Ether to Water-Insoluble, Water-Dispersible Polymer	Sag Resistance Value** (mm)
	Water-Soluble Cellulose Ether	Water-Insoluble, Water-Dispersible Polymer		
5	Hydroxypropyl methylcellulose	methylcellulose	1:1	1.5
6	"	"	1:1	1.0
7	"	"	1:1	0
8	"	"	1:1	3.0
B	*Hydroxypropyl methylcellulose	N/A	N/A	off the block

*Control

**See page 15

**Sag-Resistance Value

Sag resistance is measured by applying the sample mortar onto a concrete block using a square and thick glass template. The mortar should be screened using cuts in a right-to-left direction and then in similar cuts a left-to-right direction. Remove the glass from the mortar and center a tile in a mortar pad with the ribs in a vertical position. Then place the tile application jig into the tile. A 130 g weight is dropped from a height of 4 inches in four positions in the tile. The tile application jig is removed and the position of the tile is marked. The assembly is positioned so that the tile and mortar are in a vertical plane. After 1 hour, the sag is measured at all four corners of the tile. the "sag value" is the average of all four measurements.

Claims

1. A polymeric blend useful in a thin-bed mortar composition which comprises a water-soluble cellulose ether with a viscosity from 2,000 to 100,000 cps and a water-insoluble, water-dispersible polymer with a viscosity from 50 to 150,000 cps.

2. A composition as claimed in Claim 1 wherein the water-insoluble, water-dispersible polymer is a cellulose ether or an unneutralized polyacrylic acid.

3. A composition as claimed in Claim 2 wherein the polyacrylic acid is a copolymer of a vinyl acrylic acid monomer and from 0.05 to 2 percent crosslinking agent.

4. A composition as claimed in Claim 2 wherein the cellulose ether is methylcellulose.

5. A composition as claimed in any one of Claims 2 to 4 wherein the cellulose ether has a methoxyl content of from 10 to 25 weight percent and a hydroxypropyl content of from 0 to 6 weight percent.

6. A composition as claimed in Claim 4 wherein cellulose ether has a methoxyl content of from 18 to 23 weight percent and no hydroxypropyl content.

7. A composition as claimed in Claim 1 wherein the water-soluble cellulose ether is hydroxypropyl methylcellulose, methylcellulose, ethylhydroxyethylcellulose, hydroxyethylmethylcellulose or hydroxyethylcellulose.

8. A composition as claimed in Claim 7 wherein the methoxyl content of the cellulose ether is from 16 to 32 weight percent and the hydroxypropyl content of the cellulose ether is from 4 to 32 weight percent.

9. A composition as claimed in any one of the preceding claims wherein the ratio of water-soluble cellulose ether to water-insoluble, water-dispersible polymer is from 3:1 to 1:3.

10. A mortar composition comprising a water soluble cellulose ether having a viscosity of from 2,000 to 100,000 cps, and a water-insoluble water dispersible polymer with a viscosity of from 50 to 150,000 cps wherein the total amount of the said cellulose ether and polymer is from 0.2 to 2 percent by weight of the dry components of the mortar.

11. A mortar composition which comprises:-

(a) from 25 to 97 parts by weight Portland cement,
(b) from 0 to 75 parts by weight filler,
(c) a water-soluble cellulose ether with a viscosity of from 2,000 to 100,000 cps and
(d) a water-soluble water dispersible polymer with a viscosity of from 50 to 150,000 cps; wherein

the total amount of components (c) and (d) is from 0.2 to 2 parts by weight.

12. A composition as claimed in Claim 11 which comprises:-(a) from 50 to 80 parts by weight Portland cement,
(b) from 20 to 50 parts by weight filler,
wherein the amount of components (c) and (d) is from 0.2 to 0.5 parts by weight.

13. A method of improving the sag resistance of a mortar composition, which method comprises adding to the composition a water soluble cellulose ether having a viscosity of from 2,000 to 100,000 cps and a water-insoluble water-dispersible polymer having a viscosity of from 50 to 150 cps.